



TITLE:

On the Drawing of Polyvinyl Alcohol Filaments

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35. Acetylation of Polyvinyl Alcohol and Deacetylation of Polyvinyl-acetate

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Acetylation of polyvinyl alcohol and deacetylation of polyvinyl acetate with acetic acid-water have been investigated using hydrochloric acid as a catalyzer. It has been found, as shown in this following table, that from the both side of the reaction i. e. acetylation of polyvinyl alcohol and deacetylation of polyvinyl acetate, the same degree of acetylation can be attained so far as the ratios of acetic acid to water remains constant.

Acetylation of polyvinyl alconol and deacetylation of polyvinyl acetate at 40°C.						
Composition of the reaction medium	Water	vol %	50	32	24	17
	acetic acid	vol %	50	68	76	83
Equilibrium degree of acetylation in mol. %.	by acetylation		22.0	39.8	49.6	60.0
	by deacetylation		22.5	39.3	51.3	61.6

The reaction proceeded in a homogeneous system and equilibrium constant can be calculated from the above data.

36. On the Drawing of Polyvinyl Alcohol Filaments

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Polyvinyl alcohol mono-filaments have been prepared by semi-melt spinning process from concentrated aqueous solution. The mono-filaments have been stored and aged under various conditions and drawn at 50°C and 100°C. The swelling properties of the drawn filaments have been measured.

Degree of swelling decreased with increasing drawing. When water content of filaments is higher, the effect of temperature of drawing on the swelling is less remarkable. In the case of aging in closed vessels, the effect of aging is more remarkable by the drawing of lower degree. After 24 hrs. the duration of aging shows practically no effect on swelling. The optimum condition to obtain filaments of minimum swelling are as following: aging <1 hr., drawing at 100°C. Degree of swelling in water at 30°C is about 40 % of the dry weight.

Viscosity of dilute aqueous solutions of drawn filaments have been also determined. It is noteworthy that the intrinsic viscosity increases with increasing

drawing.

Some determination of contraction of filament through wet and dry heat have also been carried out. By wet heat at 90°C filament drawn to nine times of its original length contracts almost completely to its original length. By dry heat at 250°C filament drawn as high as five times, contracts completely.

37. On the Heat Decomposition of Polyvinyl Alcohol Fiber

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X-ray investigation of the process of heat decomposition of polyvinyl alcohol (P. V. A.) fiber have been carried out. After the heat treatment (200°C, 7 min.) P. V. A. were dried (110°C, 24 hrs.) and subjected to heat decomposition in an electric oven. The oven was heated from 150°C, and 415°C, and during the heating fibers were not allowed to contract. The process may be divide into four stages. 1), growth of crystallites between 150° and 200°C. 2), growth of crystallites and their disorientation between 200° and 250°C. 3), melting of crystallites and decomposition between 250° and 350°C. 4), carbonization above 350°C. Rate of the weight loss of the sample due to dehydration was the greatest between 250° and 300°C.

38. Determination Carboxylic Groups in Vinylon Fiber

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Determination of carboxylic groups of Vinylon fiber have been carried out according to the method of reversible methylene-blue absorption. Polyvinyl alcohol (P. V. A.) fiber is subjected to heat-treatment (5 min. at ca. 215°C), then partially (35-40 mol %.) formalized so Vinylon is obtained. As is shown in table 1, various pretreatment gives different results, so we cannot determine the true contents of the carboxylic groups of the Vinylon fibr. But it seems, that this method is useful for the technical characterization of Vinylon fiber.

This table 2 shows M. B. absorption of Vinylon after bleaching.